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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON, D.C. 20546

REPLY TO ATTN OF: GP

NOV 5 1973

TO: KSI/Scientific & Technical Information Division
Attention: Miss Winnie M. Morgan

FROM: GP/Office of Assistant General Counsel for Patent Matters

SUBJECT: Announcement of NASA-Owned U.S. Patents in STAR

In accordance with the procedures agreed upon by Code GP and Code KSI, the attached NASA-owned U.S. Patent is being forwarded for abstracting and announcement in NASA STAR.

The following information is provided:

U.S. Patent No.	: 3,765,958
Government or Corporate Employee	: Government
Supplementary Corporate Source (if applicable)	: ~
NASA Patent Case No.	: LEW-10805-3

NOTE - If this patent covers an invention made by a corporate employee of a NASA Contractor, the following is applicable:
Yes [ ] No [X]

Pursuant to Section 305(a) of the National Aeronautics and Space Act, the name of the Administrator of NASA appears on the first page of the patent; however, the name of the actual inventor (author) appears at the heading of column No. 1 of the Specification, following the words ". . . with respect to an invention of . . ."

Elizabeth A. Carter

Elizabeth A. Carter
Enclosure
Copy of Patent cited above

(NASA-Case-LEW-10805-3) METHOD OF HEAT TREATING A FORMED POWDER PRODUCT MATERIAL Patent (NASA) 5 p CSCL 11F
N74-10521
Unclas
00/17 20122

United States Patent [19]

Freche et al.

[11] **3,765,958**

[45] **Oct. 16, 1973**

[54] **METHOD OF HEAT TREATING A FORMED POWDER PRODUCT MATERIAL**

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[73] Assignee: **The United States of America as represented by the Administrator of the National Aeronautics of Space Administration,** Washington, D.C.

[22] Filed: **June 28, 1972**

[21] Appl. No.: **266,928**

Related U.S. Application Data

[62] Division of Ser. No. 29,917, April 20, 1970, Pat. No. 3,702,791.

[52] **U.S. Cl.** **148/126,** 29/420.5, 75/200, 75/226

[51] **Int. Cl.** **B22f 3/24**

[58] **Field of Search** 75/226, 200, 214; 148/126; 29/420.5

[56] **References Cited**

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[57] **ABSTRACT**

Heat treating a product material of prealloyed powders after shaping by superplastic deformation restores the ability of the material to resist deformation at high temperatures. Heat treating is accomplished by heating to a temperature between the solidus and liquidus with the application of isostatic pressure to close any voids. This pressure may be simultaneously applied while the material is at the heat treating temperature. The pressure may also be applied when the material cools to a temperature between that at which it is shaped and the solidus.

6 Claims, 3 Drawing Figures

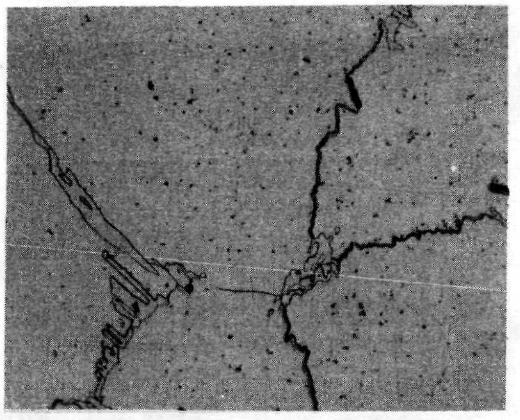

FIG. 3

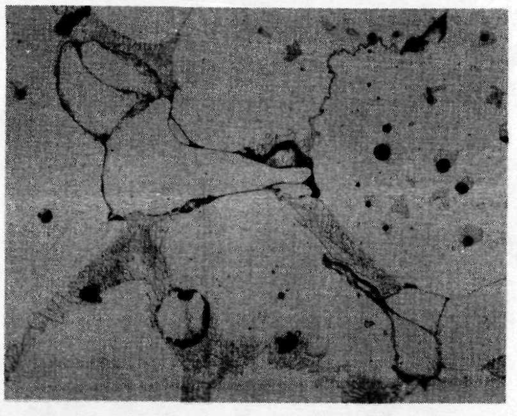

FIG. 2

FIG. 1

METHOD OF HEAT TREATING A FORMED POWDER PRODUCT MATERIAL

RELATED APPLICATION

This application is a division of copending application Ser. No. 29,917 filed Apr. 20, 1970 and now patent No. 3,702,791.

ORIGIN OF THE INVENTION

The invention described herein was made by employees of the United States Government and may be manufactured and used by or for the Government for governmental purposes without the payment of any royalties thereon or therefor.

BACKGROUND OF THE INVENTION

This invention is concerned with heat treating superalloy powder product meterials. The invention is particularly directed to improving powder materials having an ultrafine grain size for superplastic behavior at high temperatures by heat treating to increase the grain size in the final product.

Conventionally cast and wrought alloys are utilized for the hot components of gas-turbine engines. Cast nickel-base alloys are generally used for turbine buckets and stator vanes, whereas wrought alloys are used for turbine discs. In more advanced engines wrought nickel-base alloys are also used for compressor discs and blades in the latter compressor stages.

The operating cycle temperatures must be raised in advanced engines to meet the demand for increased performance. Nickel-base alloys that can be used at high temperatures throughout the engine have been suggested. However, most high strength nickel-base alloys are highly alloyed and metallurgically very complex. As a consequence, severe macro- and micro-segregation can occur in castings, such as turbine buckets and stator vanes, so that the full-strength potential of the alloy is not realized. Also, in ingots, the usual starting stock for breakdown operations, segregation increases the difficulty of forming the alloys.

SUMMARY OF THE INVENTION

Fine prealloyed powders of highly alloyed superalloy compositions may be consolidated and then shaped in separate steps. Likewise these powders may be consolidated and shaped simultaneously. During the shaping operation the consolidated powders are heated to temperatures at which the material exhibits superplastic behavior, and only relatively low pressures need be applied to shape the material. At intermediate temperatures significant increases in strength over the cast or wrought counterparts of the alloyed material can be obtained by alloys consolidated or shaped in accordance with the invention.

Suitable heat treatments are utilized to improve elevated temperature properties compared to the cast or wrought counterparts of the alloys. Heat treating at temperatures between the solidus and liquidus with a simultaneous imposition of isostatic pressure is used to obtain a suitably coarse microstructure and a solidification structure resulting from partial melting at the grain boundaries that will provide good high temperature strength and a structure free from voids. In an alternate embodiment the isostatic pressure is applied at a temperature between the shaping temperature and the solidus.

OBJECTS OF THE INVENTION

It is, therefore, an object of the present invention to heat treat articles of manufacture made from superalloy compositions which are too highly alloyed to be cast without gross segregation.

A further object of the invention is to provide a method of heat treating a superalloy article of manufacture in which significant deformation has been achieved with relatively low applied forces.

A still further object of the invention is to provide a method of making a superalloy article of manufacture in which the microstructure of the formed material is coarsened by heat treating at temperatures above the incipient melting point of the alloy without void formation.

Another object of the invention is to provide superalloys having higher strength at intermediate or at high temperatures than can be obtained by conventional cast and cast-wrought processes.

These and other objects of the invention will be apparent from the specification which follows and from the drawing.

DESCRIPTION OF THE DRAWING

The figures are micrographs of HS-31 powder product at a magnification of **500**.

FIG. **1** shows the as-extruded alloy,

FIG. **2** shows the material after the first step of heat treatment, and

FIG. **3** shows the material after final heat treatment with the application of pressure.

DESCRIPTION OF THE PREFERRED EMBODIMENT

The method of the present invention utilizes prealloyed powders of a highly alloyed superalloy composition. The powders are extremely fine and have a high purity.

The powders can be prepared by inert gas atomization or other methods, such as the rotating electrode method. To produce the powders by atomization remelt stock is first melted under an inert gas or in vacuum in an induction furnace, and the melt is then atomized under an inert gas. The resulting powders are screened, and only suitable size powders are used for further processing in accordance with the invention.

The prealloyed powders are then consolidated in the form of either bar stock or preforms. More particularly, the prealloyed powders can be made into bar stock by extrusion, or by a combination of hot pressing and extrusion. These powders can also be made into preforms for turbine buckets or other desired articles of manufacture. These preforms are made by slip-casting, by pressing in a shaped die, or by enclosing the powder in a suitably shaped container, such as a metallic can.

The consolidated powders are then heated to a temperature at which the material exhibits superplastic behavior. While a blank or preformed shape of these consolidated powders is still hot, pressure is applied to form the powders into the desired configuration. This pressure may be applied unidirectionally to suitably shaped dies.

Because of the superplastic behavior of the material a very low pressure is required to shape the consolidated powders. The shaping may be accomplished at pressures as low as 1,000 psi.

An important feature of the invention is that the formed part or article of manufacture is then heat treated to obtain suitably coarse microstructures for superior high temperature strength. Isostatic pressure is imposed during heat treating and the temperature may be either above the incipient melting point or between the forming temperature and the solidus of the powder material.

EXAMPLES

In order to better illustrate the invention test samples of an experimental nickel-base superalloy were prepared and tested. The nominal composition of the alloy is shown in TABLE 1.

TABLE I — NOMINAL COMPOSITION OF ALLOY

Element	wt.%
Tantalum	8
Tungsten	4
Molybdenum	4
CColumbium	2.5
Chromium	6
Aluminum	6
Zirconium	0.75
Carbon	0.125
Nickel	Balance

This cast nickel-base superalloy was melted under vacuum in an induction furnace. The melt was atomized under argon to spheroidal powders which were screened with Tyler screens to —60 mesh. Only the —60 mesh fraction was used for further processing. The sieve analyses for the —60 mesh fraction for the alloy is shown in TABLE II.

TABLE II — PARTICLE SIZE DISTRIBUTION OF ATOMIZED POWDER

Tyler screen size	Percent
60/100	5.0
100/500	13.5
150/270	30.0
270/325	7.0
325/400	9.0
400	35.5

These fine powders were sealed in evacuated mild steel cans. The canned powders were heated to 2,200° F in a furnace and transferred to an extrusion press. Here the powders were extruded into bars and the cans were reduced in size from 2 inches to approximately 9/16 inch in diameter by passing them through an extrusion die.

The bars were first tested in the as-extruded condition. The nickel-base alloy had an elongation of more than 600% after testing at 1,900° F and 1,000 psi for 4.1 hours. These very high elongations which occurred in elevated temperature tensile and stress rupture tests indicated superplastic behavior in this temperature region.

Samples of the as-extruded powder product of the alloy were upset and formed to shapes in closed dies to show that the material can be formed in compression to take advantage of this superplastic behavior. A hydraulically operated press with an in-place graphite susceptor induction heating furnace was used. Bar specimens approximately ⅝ inch high were heated to 2,000° F and pressed. Pressure was applied to the circular ends of the specimens through high temperature alloy plates which were heated to the same temperature as the specimen. An initial load of 155 pounds was applied. The load was increased as necessary to maintain a relatively constant strain rate of between 0.03 to 0.07 inch per inch per minute. This strain rate was used to approximate the rate observed when superplasticity was encountered with the alloy in a stress rupture test. The upset specimen had a diameter of 1.1 inch and a thickness of 0.175 inch after pressing.

According to the present invention heat treatments to effect solutioning and aging were performed in vacuum or under argon on unmachined extruded bars of the nickel-base alloy. These heat treatments coarsened the microstructure of extruded powder products and substantially improved stress rupture life for the alloy compared to the life of the as-extruded powder product at an intermediate temperature. At 1,200° F and 105,000 psi the extruded and heat-treated powder product of the alloy had a rupture life of 975 hours, whereas the as-extruded powder product had a life of 374 hours.

Heat-treated extruded samples of the alloy had substantially lower rupture life at high temperatures of 1,800° to 2000° F than as-cast samples. For the heat treated, extruded powder product it was 2.2 against 90 hours at 1,900° F and 15,000 psi.

By simultaneously applying pressure and heating above the incipient melting point of the as-extruded nickel-base alloy powder product void formation is prevented. This product was successfully heated to 2,400° F, which is about 50° above the incipient melting point, under a pressure of 10,000 psi. This simultaneous application of pressure and the high temperature coarsened the microstructure to a greater degree than by conventional heat treatments.

Test samples of a commerical cobalt-base alloy were also prepared and tested to illustrate the beneficial effect of the heat treatment that utilized both high temperatures above the incipient melting point and high pressures. The cobalt-base alloy identified as HS-31 was made by the aforementioned preålloyed powder process. The microstructure of as-extruded HS-31 powder product is shown in FIG. 1.

As extruded bars of the cobalt-base alloy were heat treated for 1 hour at 2,400° F at atmospheric pressure. The microstructure after this heat treatment is shown in FIG. 2. This is about 60° F above the incipient melting point.

The grain growth was accompanied by the formation of large voids. Subsequent application of isostatic pressure of 30,000 psi at 2200° F grew the grains further and closed the voids. This restored the integrity of the samples as shown in FIG. 3. Operation at 13,000 psi and 1,800° F resulted in a 20 hour life, which is double that of the as-cast alloy. Operations at 61,000 psi and 1,200° F resulted in a 420 hour life compared to 10 hours for the cast alloy.

What is claimed is:

1. A method of heat treating a dense formed powder product material selected from the group consisting of nickel and cobalt superalloys to coarsen the microstructure thereof comprising the steps of

heating the material to a temperature between the solidus and liquidus of the formed powdered material thereby promoting grain growth and void formation, and

imposing isostatic pressure on the material to close said voids.

2. A method of heat treating as claimed in claim 1 wherein the isostatic pressure is imposed simultaneously with the heating.

3. A method of heat treating as claimed in claim 1 including the step of

cooling the material to a second temperature below the incipient melting point before imposing the isostatic pressure, said isostatic pressure being applied at said second temperature.

4. A method of heat treating as claimed in claim 4

wherein the material is cooled to a temperature between that at which the material exhibits superplastic behavior and the solidus temperature.

5. A method of heat treating as claimed in claim 1 wherein the formed powder product material is a nickel-base alloy.

6. A method of heat treating as claimed in claim 1 wherein the formed powder product material is a cobalt-base alloy.

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